
फिप्रोनिल, तकनीकी — विशिष्टि

Fipronil, Technical — Specification

ICS 65.100.10

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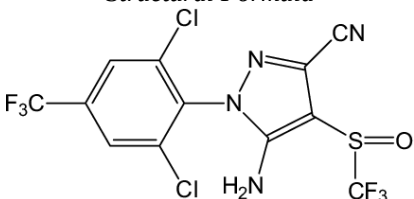
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Fipronil technical is used in the preparation of insecticidal formulations for environmental/agricultural/health purpose uses.

Fipronil is the accepted common name by the international organization for standardization for 5-Amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)sulfinyl]-1H-pyrazole-3-carbonitrile.

The empirical and structural formulae and the molecular mass of fipronil are given below:

Empirical Formula	Structural Formula	Molecular Mass
$C_{12}H_4Cl_2F_6N_4OS$		437.14

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticide Act*, 1968, and the rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and rules, wherever applicable.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

FIPRONIL, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for fipronil, technical.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	Title
IS 1070 : 2023	Reagent grade water — Specification (fourth revision)
IS 6940 : 1982	Methods of test for pesticides and their formulations (first revision)
IS 8190 (Part 1) : 1988	Requirement for packing of pesticides: Part 1 Solid pesticides (second revision)
IS 10946 : 1996	Methods of sampling for technical grade pesticides (first revision)

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of white to beige coloured lumps or powder with characteristic smell. It shall be devoid of hard aggregates and free of extraneous matter.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in LDPE/HDPE sack of thickness not less than 0.125 mm. The capacity of the LDPE/HDPE sack shall be 25 kg, 50 kg, and 100 kg. This shall be individually packed in fiber board drum

with metal on the base and the lid. The capacity of the fiber board drum shall be 25 kg, 50 kg, and 100 kg only. The container shall also meet the general requirements given in 8190 (Part 1).

5 MARKING

5.1 The sack shall bear legibly and indelibly the following:

- Name of the material;
- Name and address of the manufacturer;
- Batch number;
- Date of manufacture;
- Date of expiry;
- Net quantity;
- Nominal Fipronil content, percent (*m/m*);
- Cautionary notice as worded in the *Insecticides Act*, 1968, and rules framed thereunder; and
- Any other information required under the (*Legal Metrology Packaged Commodities Rules*, 2011).

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10946.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col (4) of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (see IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Fipronil, Technical*(Clause 3.2)*

Sl No.	Characteristic	Requirement	Method of Test, Ref. to
(1)	(2)	(3)	(4)
i)	Fipronil contents, percent by mass, <i>Min</i>	90.0	Annex A
ii)	Moisture content, percent by mass, <i>Max</i>	1.0	IS 6940
iii)	Acidity (as H ₂ SO ₄), percent by mass, <i>Max</i>	0.5	IS 6940
	or	0.5	
	Alkalinity as NaOH, percent by mass, <i>Max</i>		

ANNEX A

[Table 1, SI No. (i)]

DETERMINATION OF FIPRONIL CONTENT

A-1 PRINCIPLE

The fipronil content is determined by high performance liquid chromatography (HPLC) equipped with UV-VIS detector using internal standard technique.

A-2 APPARATUS

A-2.1 A high performance liquid chromatography system equipped with ultraviolet detector and chromatographic software/printer plotter-cum-integrator is used for this determination. The suggested operative parameters are as follows, but can be changed, if necessary, provided standardization is done:

Column	Stainless steel, 25 cm × 4.61 mm i.d., packed with 5 µm Cosmosil packed, RP 18
Temperature	Ambient
Mobile phase	Acetonitrile : Water (80 : 20 v/v)
Flow rate	0.5 ml/min
Injection volume	20 µl
Detector	Ultraviolet detector (280 nm)

A typical HPLC chromatograms of fipronil, technical are given in Fig. 1 and Fig. 2.

A-2.2 Analytical Balance

A-2.3 Microlitre Syringe — 25 µl capacity

A-2.4 Magnetic Stirrer

A-2.5 Ultrasonic Bath

A-2.6 Standard Glassware

A-3 REAGENTS

A-3.1 Fipronil Reference Standard — of known purity

A-3.2 Acetonitrile — HPLC/AR grade

A-3.3 Water — HPLC/AR grade

A-3.4 Acetophenone (Internal Standard) — of known purity

A-4 PROCEDURE

A-4.1 Preparation of Mobile Phase

Mix acetonitrile and water in the proportion of 80 : 20 v/v and pass through membrane filter under vacuum. Homogenize the mixture using a magnetic stirrer and allow to attain room temperature.

NOTE — Dissolution of acetonitrile in water is endothermic, hence allow the mixing to start at ambient temperature and continue till it attains room temperature.

A-4.2 Preparation of Internal Standard Solution

Weigh accurately 0.1 g acetophenone (99.0 percent) into a 25 ml (Concentration 4 mg per ml) volumetric flask. Dissolve this in acetonitrile and ultrasonicate for 10 minutes. Make up to volume with acetonitrile.

A-4.3 Preparation of Standard Solution

Weigh accurately 0.1 g of fipronil analytical standard of known purity into 100 ml volumetric flask. Add 50 ml of acetonitrile and dissolve content manually by shaking and sonicate for 15 minutes. Allow it to attain room temperature and make up the volume up to the mark with acetonitrile. Call this as Solution “A”. (conc. 1 mg per ml)

Pipette out 10 ml solution “A” into a 100 ml volumetric flask and make up the volume upto the mark with acetonitrile. Shake well to homogenize. Call this as Solution “B”.

Pipette out 5.0 ml of Solution “B” into a 50 ml volumetric flask and add 5 ml Internal Standard solution by pipette and make up the volume upto the mark with mobile phase and homogenize. Call this as Solution “C”.

A-4.4 Preparation of Sample Solution

Weigh accurately 0.1 g of previously dried (for 4 h at 105 °C) fipronil technical into a 100 ml volumetric flask and proceed as in A-4.3.

A-5 ESTIMATION

5.1 Inject 20 µl standard solutions until the area quotients of reference standard solution/ sample solution of two successive chromatograms do not deviate from each other by more than 2 percent.

Then use the following injection sequence:

... C1 S1 C2S1 C1 S2 C2 S2.....

where

C = standard solution, and
S = sample solution (1, 2..., n).

A-5.2 Retention Time (Guide Values)

Fipronil = 8.1 min (approximately)
Acetophenone (IS) = 4.4 min (approximately)
Total run time = 10 min.

A-6 CALCULATION

A-6.1 Fipronil content, percent by mass =

$$\frac{A_2 \times M_1 \times I_2 \times P}{A_1 \times M_2 \times I_1}$$

where

A_2 = peak area of fipronil in sample;
 M_1 = mass, in g, of fipronil in standard solution;
 I_2 = peak area of internal standard in fipronil sample solution;
 P = known purity of fipronil reference standard;
 A_1 = peak area of fipronil in standard;
 M_2 = mass, in g, of the sample taken for the test; and
 I_1 = peak area of internal standard in fipronil standard solution.

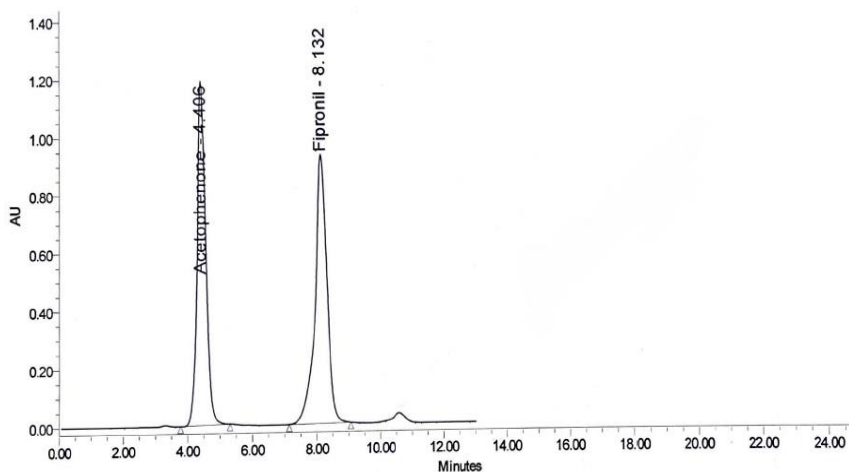


FIG. 1 A TYPICAL CHROMATOGRAM FOR STANDARD

Standard $R_f = 1.08$

	Peak Name	RT	Area	Percent Area	Height
i)	Acetophenone	4.406	22 260 716	48.21	1 192 782
ii)	Fipronil	8.132	23 915 969	51.79	935 238

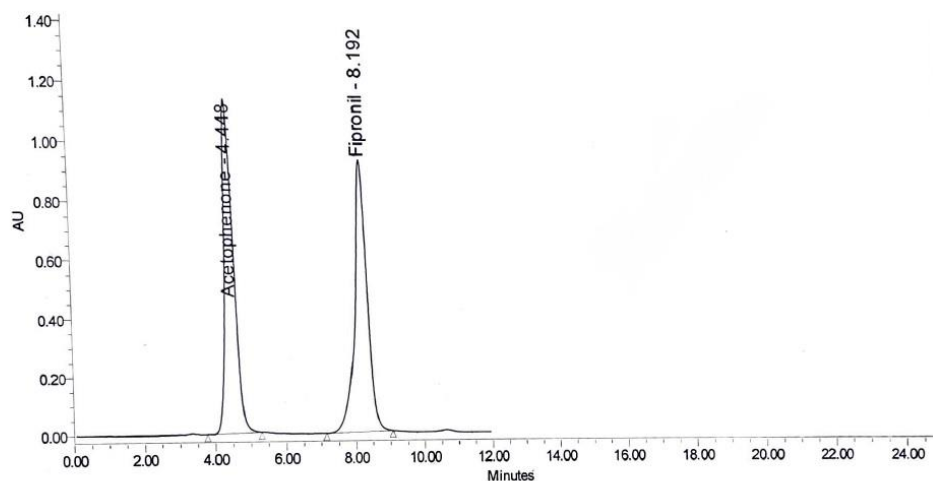


FIG. 2 A TYPICAL CHROMATOGRAM FOR STANDARD FOR SAMPLE

Sample $R_f = 1.10$

	Peak Name	RT	Area	Percent Area	Height
i)	Acetophenone	4.448	21 623 150	47.60	1 140 890
ii)	Fipronil	8.192	23 800 997	52.40	939 580

ANNEX B*(Foreword)***COMMITTEE COMPOSITION**

Pesticides Sectional Committee, FAD 01

<i>Organization</i>	<i>Representative(s)</i>
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR J. P. SINGH (<i>Chairperson</i>)
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (<i>Alternate</i>)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (<i>Alternate</i>)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (<i>Alternate</i>)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR M. S. KAMATH (<i>Alternate</i>)
Crop Care Federation of India, New Delhi	DR J. C. MAJUMDAR
Crop Life India, New Delhi	SHRI ASITAVA SEN MS NIRUPAMA SHARMA (<i>Alternate</i>)
CSIR - Indian Institute of Toxicology Research, Lucknow	DIRECTOR DR SHEELENDRA P. SINGH (<i>Alternate</i>)
Food Safety and Standards Authority of India, New Delhi	ADVISOR (STANDARDS)
FMC India Pvt Limited, Bengaluru	SHRI CHIRAG PATEL
IDMA Laboratories Limited, Chandigarh	DR INDRA RAI
Indian Agricultural Research Institute, New Delhi	DIRECTOR
Indian Institute of Packaging, Mumbai	DR TANWEER ALAM
Indian Pest Control Association, New Delhi	SHRI UDAYAN GHOSH
Institute of Pesticide Formulation Technology, Gurugram	DR M. VAIRAMANI
Ministry of Agriculture, Department of Agriculture, Chennai	JOINT DIRECTOR OF AGRICULTURE (RES.) DEPUTY DIRECTOR LAB (<i>Alternate</i>)
National Centre for Integrated Pest Management, New Delhi	DR SUMITRA ARORA
National Institute of Plant Health Management, Hyderabad	DR MAHESH SAINI MS T. SRIDEVI (<i>Alternate</i>)

<i>Organization</i>	<i>Representative(s)</i>
Pesticide Manufactures and Formulators Association of India (PMFAI), Mumbai	DR ARCHANA SRIVASTAVA DR UDAY KUMAR (<i>Alternate</i>)
Regional Pesticides Testing Laboratory, Chandigarh	DR ROHIT SINGH
In Personal Capacity (4-6-90/2/8/2, Sri Devi Nilayam Tejaswinagar Coloni, Attapur, Hyderabad – 500048)	SHRI C. V. RAO
In Personal Capacity (263, Sector 28, Faridabad – 121008)	SHRI VIPIN SAINI
BIS Directorate General	SHRIMATI SUNEETI TOTEJA, SCIENTIST 'E'/DIRECTOR AND HEAD (FOOD AND AGRICULTURE) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

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Amendments Issued Since Publication

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